



Convention Date (Canada): Jan. 7, 1941.

549,931

Application Date (in United Kingdom): June 10, 1941. No. 7333/41.

Complete Specification Accepted: Dec. 15, 1942.

## COMPLETE SPECIFICATION.

## The Recovery of Tocopherol (Vitamin E) and Valuable By-Products from Vegetable Oils.

We, THE OGILVIE FLOUR MILLS COMPANY LIMITED, of City of Montreal, Province of Quebec, Dominion of Canada, a Canadian Company, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to improvements in the treatment of saponifiable oils and it is especially concerned with the recovery therefrom of tocopherol (Vitamin E) in relatively concentrated form. Prior methods of concentrating tocopherol from wheat germ and similar oils have all included alkaline saponification. This is objectionable because tocopherol is known to be unstable in alkaline solutions while a further difficulty encountered arises from the presence of sterols and carotinoid pigments which are difficult to remove without loss of tocopherol.

According to the present invention the improved method of receiving tocopherol (Vitamin E) from vegetable oils comprises treating them dissolved in an organic solvent with cold concentrated sulphuric acid, and then separating the organic solvent-phase and evaporating the solvent. This leaves a clear, colorless oil, free from sterols and which contains from 5 to 10 times the tocopherol content of the original oil.

It is advisable to wash the separated solvent-phase before evaporating the solvent with dilute alkali in order to remove the traces of sulphuric acid and sulphonated compounds which may be present. The preferred solvent to use is petroleum ether, although other inert solvents which do not readily react with sulphuric acid, such as acetone, may likewise be employed. The concentration of the sulphuric acid should be from 85 to 88%.

The separated acid phase may be diluted with water and the resulting white precipitate filtered and washed free of acid. The residue so secured has the properties of a detergent and it can be extracted with ethyl ether to yield an oil having anti-oxidant properties.

[Price 1/-]

The sulphuric acid treatment removes up to 90% of the oil and it is especially applicable to wheat germ oil. The tocopherol-containing extract is free from sterols and carotinoid pigments and it does not respond to the Liebermann-Bouchard reaction for sterols nor the Carr - Price reaction for carotinoid pigments.

## EXAMPLE.

40 grams of wheat germ oil are dissolved in 1 litre of petroleum ether and 200 cc. of 85% sulphuric acid are added slowly and in a fine stream under constant agitation. The agitation is continued for 5 minutes after the addition of the sulphuric acid has been completed and a reddish-brown mass settles to the bottom of the container. The clear supernatant petroleum ether phase is then decanted and transferred to a distilling apparatus from which the petroleum ether is removed by distillation. The resulting product is a clear, colorless oil which gives no tests for sterols nor for carotinoid pigments but which gives the tests for tocopherol.

The acid phase is treated with a large excess of water, whereupon the red-brown color disappears and the sludge is flocculated as a white precipitate. It is removed by filtration and washed free of excess acid to yield an effective detergent. Upon extraction with ethyl ether an oil is recovered which readily emulsifies with water, which on standing develops an odor of linolool and possesses strong anti-oxidant properties.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. An improved method of recovering tocopherol (Vitamin E) from vegetable oils which comprises treating a solution of vegetable oil in an organic solvent with cold concentrated sulphuric acid, separating the organic solvent-phase and then evaporating the solvent, to leave an oil which is rich in tocopherol content.

2. An improved method according to

W. 43 25

claim 1 in which the separated solvent phase is washed with dilute alkali before the solvent is evaporated.

5 3. An improved method according to claim 1 or 2, in which the solvent employed is petroleum ether.

4. An improved method according to any of the preceding claims in which 85% to 88% sulphuric acid is used.

10 5. An improved method according to any of the preceding claims in which the separated acid phase is diluted with water and the resulting white precipitate filtered and washed free of acid.

15 6. An improved method according to any of the preceding claims in which wheat germ oil is dissolved in petroleum ether and the cold concentrated sulphuric acid added to it under agitation, then the mixture is allowed to settle and the  
20 petroleum ether fraction is decanted,

washed with dilute alkali and then distilled to remove the petroleum ether and to leave a clear, colorless, sterol-free oil which is relatively rich in tocopherol, the acid phase being diluted with water, and the resulting precipitate filtered off and extracted with ethyl ether to yield an oil having anti-oxidant properties and to leave a residue having detergent  
25 30 properties.

7. The improved method of refining the unsaponifiable fractions of vegetable oils substantially as hereinbefore described.

8. The products when secured as a result of the improved method of recovering tocopherol (Vitamin E) from vegetable oils as hereinbefore particularly described and ascertained.  
35

Dated this 10th day of June, 1941.

MARKS & CLERK.

Abingdon : Printed for His Majesty's Stationery Office, by Burgess & Son.

[Wt. 8229A.—12/1946.]